Diamond-like carbon (DLC) coatings are a new environmentally friendly form of low-friction, high wear resistance, and self-lubricating solid material which could eliminate the need for grease-based lubrication in some applications. Certain DLC coatings can often adhere poorly to various substrates, so interlayers are sometimes used, such as silicon. However, some substrates still have poor adhesion to DLC at micron-level coating thicknesses even with the interlayer. The ratio of modulus to hardness, sp²/sp³ hybridization of the carbon in the coating, and surface morphology have all been found to be factors affecting adhesion.

Objective
To investigate the relationships between coating thickness, modulus, hardness, hybridization, and surface morphology to DLC adhesion.

Experimental Procedure

Samples
Four thicknesses of DLC coating were grown onto five different substrates with a silicon interlayer. These five substrates included 17-4PH steel, 13-8PH steel, S118, D2 and P20. The thicknesses were analyzed to determine an optimal DLC coating process.

Raman Spectroscopy (HORIBA Scientific T64000)
- Hydrogenated Diamond-like carbon (DLC) consists of an amorphous carbon matrix of diamond-like sp² orbitals and graphite-like sp² orbitals, and includes hydrogen bonds embedded within the matrix.
- DLC coatings are grown onto materials through Plasma Assisted Chemical Vapor Deposition (PACVD). The material was put into a vacuum chamber where it was sputter cleaned, nitrided and then coated with a silicon interlayer. Finally a carbon-rich gas was introduced into the chamber and ignited into a plasma to deposit a DLC coating.

Nanoindentation (Hysitron TI-950 TriboIndenter)
- Adhesion failure identified through inconsistencies in the scratch graphs and quantified through micrographs of the scratches.
- 3 distance controlled scratches of length 10 μm for each substrate.
- 5 μm conical tip with a high load head

Atomic Force Microscopy (AFM)
- Surface roughness and morphology analyzed.

Results & Discussion

Raman Spectroscopy

Table 1. Raman spectrum of two DLC samples (17-4PH & 13-8PH) indicates a compositional range of 35-45% for the sp² content using the peaks intensity ratio (I(D)/I(G)). Limitations in the Raman shift range in the T64000 during scanning are reflected in data collection as only differences between spectra were considered with I(D)/I(G) of 0.86 indicating higher sp² content than (0.9). High-low designation is applied. In Figure 3 increased Beta 4 coating thicknesses in both (b) and (d) compared to Beta 1 in (a) and (c) indicates a lower sp² content and higher G dispersion as can be seen in Table 1. Beta 2 samples overall have the highest diamond content and 13-8PH have higher sp² content in both Beta 2 and Beta 3 thicknesses.

Table 2. Diamond-like carbon coatings exhibit good adhesion to the substrates as shown in the scratch test adhesion results.

Scratch Adhesion

Nanoindentation

Microhardness (GPa)

Table 3. Hardness of samples from nanoindentation testing with a 95% confidence interval.

Conclusion & Recommendations

Beta 3 coatings are the best candidate for dry lubrication applications due to their lack of failure during scratch adhesion and intermediate roughness and sp² content. Beta 2 has the highest sp² content and hardness, but Beta 3 adheres better to the substrates. In the future, we recommend investigating a set with a thickness range of 7-10 μm to achieve the best properties of both coatings. We also recommend using XPS to confirm hybridization and REELS to confirm hydrogen content. It would be beneficial to cut cross-sections of the samples and analyze any interlayer interactions along with quantifying the actual thickness of the DLC coating as well. Correlation measurements should be taken before and after the PACVD process to measure residual stress. If scratch adhesion is performed again, longer scratches should be made.